

# Data Validation SOP

HW-2, Rev. 11

Metals

Evaluation of Metals Data for the Contract Laboratory Program (CLP)

based on

SOW. 3/90

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(SOP Revision XI)

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1.0 **Scope**

1.1 This procedure is applicable to inorganic data obtained from contractor laboratories working for Hazardous Waste Site Contract Laboratory Program (CLP).

1.2 The data validation is based upon analytical and quality assurance requirements specified in Statement of Work (SOW) 3/90 .

2.0 **Responsibilities** - Data reviewers will complete the following tasks as assign the Data Review Coordinator:

2.1. For a **total review**:

2.1.1 **Data Assessment - "Total Review-Inorganics" Checklist Appendix (A.1).**  
The reviewer must answer every question on the checklist.

2.1.2 **Data Assessment - Data Assessment Narrative (Appendix A.2)**  
The answer on the checklist must match the action in the narrative (appendix A.2) and on Form I's. Do not use pencil to write the narrative.

2.1.3 **Contract Non-Compliance - SMO Report (Appendix A.3)**  
This report is to be completed only when a serious contract violation is encountered, or upon the request of the Data Validation Task Monitor, or Tech Project Officer (TPO). Forward 5 copies: one each for internal files, appropriate Regional TPO, Sample Management Office (SMO) and last two address Mailing List for Data Reviewers (Appendix A.4). In other cases, all contract violations should be appended to the end of the Data Assessment Narrative (See A.2.2).

#### 2.1.4 CLP Data Assessment Summary Forms

##### 2.1.4.1 Appendix A.5

Fill in the total number of analytes analyzed by different analyses and the number of analytes rejected or flagged as estimated due to corresponding quality control criteria. Place an "X" in boxes where analyses were not performed, or criteria do not apply.

##### 2.1.4.2 Appendix A.6

Data reviewer is also required to fill out Inorganic Regional Data Assessment form (Appendix A.7) provided by EPA Headquarters. Codes listed on the form will be used to describe the Data Assessment Summary.

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2.1.5 Data Review Log: It is recommended that each data reviewer should maintain a the reviews completed to include: a. date of start of case

- b. date of completion of case review
- c. site
- d. case number
- e. contract laboratory
- f. number of samples
- g. matrix
- h. hours worked
- i. reviewer's initials

2.1.6 Telephone Record Log - the data reviewer should enter the bare facts of inquiry, before initiating any phone conversation with CLP laboratory. After the case review has been completed, mail white copy of Telephone Record Log to the laboratory and pink copy to SMO. File yellow copy in the Telephone Record Log folder, and attach a xerox copy of the Telephone Record Log to the completed Data Assessment Narrative (Appendix A.2).

##### 2.1.7 Forwarded Paperwork

- 2.1.7.1 Upon completion of review, the following are to be forwarded to the Regional Sample Control Center (RSCC) located in the Surveillance and Monitoring Branch
- a. data package
  - b. completed data assessment checklist (Appendix A.1, original)
  - c. SMO Contract Compliance Screening (CCS)
  - d. Record of Communication (copy)
  - e. CLP Reanalysis Request/Approval Record (original + 3 copies)
  - f. Appendix A.6 (original).
- 2.1.7.2 Forward 2 copies of completed Data Assessment Narrative (Appendix A.2) along with 2 copies of the Inorganic Data Assessment Form (Appendix A.6) and Telephone Record Log, if any, one each for appropriate Regional TPO, and the other one to EPA EMSL office in Las Vegas. The addresses of TPOs and office in Las Vegas are given in Appendix A-4.
- 2.1.8 **Filed Paperwork** - Upon completion of review, the following are to be filed within MMB files:
- a. Two copies of completed Data Assessment Narrative (Appendix A.2) each carry Appendix A.6.
  - b. Telephone Record Log (copy)
  - c. SMO Report (copy Appendix A-3)
  - d. CLP Reanalysis Request/Approval Record (copy)

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3.0 **Data Completeness**

Each data package is checked by a Regional Sample Control Coordinator (RSSC) completeness. A data package is assumed to be complete when all the deliverables required under the contract are present. If a data package is incomplete, the laboratory would call the laboratory for missing document(s). If the laboratory does not respond within a week, SMO and MMB coordinator of Region II will be notified

4.0 **Rejection of Data** - All values determined to be unacceptable on the Inorganic Analysis Data Sheet (Form I) must be lined over with a red pencil. As soon as review criteria causes data to be rejected, that data can be eliminated any further review or consideration.

5.0 Acceptance Criteria - In order that reviews be consistent among reviewers, acceptance criteria as stated in Appendix A.1 (pages 4-25) should be used. Additional guidance can be found in the National Inorganic Functional Guideline October 1, 1989.

6.0 SMO Contract Compliance Screening (CCS) - This is intended to aid reviewer in locating any problems, both corrected and uncorrected. However, the validation should be carried out even if CCS is not present. Resubmittals received from laboratory in response to CCS must be used by the reviewer.

7.0 Request for Reanalysis - Data reviewers must note all items of contract non-compliance within Data Assessment Narrative. If holding times and sample times have not been exceeded, TPO may request reanalysis if items of non-compliance are critical to data assessment. Requests are to be made on "CLP Re-Analysis Request/Approval Record".

8.0 Record of Communication - Provided by the Regional Sample Control Center (RSCC) indicate which data packages have been received and are ready to be reviewed.

9.0 Rounding off numbers - The data reviewer will follow the standard practice.

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	YES	NO
A.1.1 <u>Contract Compliance Screening Report (CCS)</u> - Present?	[ ]	
<u>ACTION:</u> If no, contact RSCC.		
A.1.2 <u>Record of Communication (from RSCC)</u> - Present?	[ ]	
<u>ACTION:</u> If no, request from RSCC.		

A.1.3 Trip Report - Present and complete? ☐ ☐

**ACTION:** If no, contact RSCC for trip report.

A.1.4 Sample Traffic Report - Present? ☐ ☐

Legible? ☐ ☐

**ACTION:** If no, request from Regional Sample Control Center (RSCC).

A.1.5 Cover Page - Present? ☐ ☐

Is cover page properly filled in and signed by the lab manager or the manager's designee? ☐ ☐

**ACTION:** If no, prepare Telephone Record Log, and contact laboratory.

Do numbers of samples correspond to numbers on Record of Communication? ☐ ☐

Do sample numbers on cover page agree with sample numbers on:

(a) Traffic Report Sheet? ☐ ☐

(b) Form I's? ☐ ☐

**ACTION:** If no for any of the above, contact RSCC for clarification.

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A.1.6 Form I to IX Yes No

A.1.6.1 Are all the Form I through Form IX labeled with:

Laboratory name?	[ ]	
Case/SAS number?	[ ]	___
EPA sample No.?	[ ]	
SDG No.?	[ ]	___
Contract No.?	[ ]	
Correct units?	[ ]	___
Matrix?	[ ]	___

**ACTION:** If no for any of the above, note under Contract Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.6.2 Do any computation/transcription errors exceed 10% of reported values on Forms I-IX for:

(NOTE: Check all forms against raw data.)

(a) all analytes analyzed by ICP?	[ ]	___	___
(b) all analytes analyzed by GFAA?	[ ]	___	___
(c) all analytes analyzed by AA Flame?	[ ]	___	___
(d) Mercury?	[ ]	___	___
(e) Cyanide?	[ ]		

**ACTION:** If yes, prepare Telephone Log, contact laboratory for corrected data and correct errors with red pencil and initial.



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		<u>YES</u>	<u>NO</u>
A.1.7	<b><u>Raw Data</u></b>		
A.1.7.1	Digestion Log* for flame AA/ICP (Form XIII) present?	[ ]	___
	Digestion Log for furnace AA Form XIII present?	[ ]	
	Distillation Log for mercury Form XIII present?	[ ]	
	Distillation Log for cyanides Form XIII present?	[ ]	___
	Are pH values (pH<2 for all metals, pH>12 for cyanide) present?	[ ]	___
	*Weights, dilutions and volumes used to obtain values.		
	Percent solids calculation present for soils/sediments?	[ ]	
	Are preparation dates present on sample preparation logs/bench sheets?	[ ]	___
A.1.7.2	Measurement read out record present?		
	ICP	[ ]	___
	Flame AA	[ ]	___
	Furnace AA	[ ]	___
	Mercury	[ ]	
	Cyanides	[ ]	___
A.1.7.3	Are all raw data to support all sample analyses and QC operations present?	[ ]	___
	Legible?	[ ]	___
	Properly Labeled?	[ ]	___

**ACTION:** If no for any of the above questions  
 in sections A.1.7.1 through A.1.7.3,  
 write Telephone Record Log and contact

laboratory for resubmittals.

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		<u>YES</u>	<u>NO</u>
A.1.8	<b><u>Holding Times</u></b> - (aqueous and soil samples )		
	(Examine sample traffic reports and digestion/distillation logs.)		
	Mercury analysis (28 days). . . . . exceeded?	___	[___]
	Cyanide distillation (14 days). . . . . exceeded?		[ ]
	Other Metals analysis (6 months). . . . exceeded?	___	[___]
	<p><b><u>NOTE:</u></b> Prepare a list of all samples and analytes for which holding times have been exceeded. Specify the number of days from date of collection to the date of preparation (from raw data). Attach to checklist.</p>		
	<p><b><u>ACTION:</u></b> If yes, reject (red-line) values less than Instrument Detection Limit (IDL) and flag as estimated (J) the values above IDL even though sample(s) was preserved properly.</p>		
A.1.8.2	Is pH of aqueous samples for:		
	Metals Analysis >2?	___	[___]
	Cyanides Analysis <12?	___	[___]
	<p><b><u>Action:</u></b> If yes, flag the associated metals and cyanides data as estimated.</p>		
A.1.9	<b><u>Form I (Final Data)</u></b>		
A.1.9.1	Are all Form I's present and complete?		[ ]

**ACTION:** If no, prepare telephone record log and contact laboratory for submittal.

- A.1.9.2 Are correct units (ug/l for waters and mg/kg for soils) indicated on Form I's? ☐ ☐
- Are soil sample results for each parameter corrected for percent solids? ☐ ☐
- Are all "less than IDL" values properly coded with "U"? ☐ ☐

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	<u>YES</u>	<u>NO</u>
Are the correct concentration qualifiers used with final data?	<input type="checkbox"/>	<input type="checkbox"/>

**ACTION:** If no for any of the above, prepare Telephone Record Log, and contact laboratory for corrected data.

- A.1.9.3 Are EPA sample # s and corresponding laboratory sample ID # s the same as on the Cover Page, Form I's and in the raw data? ☐ ☐
- Was a brief physical description of samples given on Form I's? ☐ ☐
- Was the dilution of any sample diluted beyond the requirements of the contract noted on Form I or Form XIV? ☐ ☐

**ACTION:** If no for any of the above, note under Contract-Problem/Non-Compliance of the "Data Assessment Narrative".

A.1.10 **Calibration**

- A.1.10.1 Is record of at least 2 point calibration

present for ICP analysis? ☐ ☐

Is record of 5 point calibration present for  
Hg analysis? ☐ ☐

Is record of 4 point calibration present for:

Flame AA? ☐ ☐

Furnace AA? ☐ ☐

Cyanides? ☐ ☐

Is one calibration standard at the CRDL level for  
all AA (except Hg) and cyanides analyses? ☐ ☐

**ACTION:** If no for any of the above, write in the  
Contract Problem/Non-Compliance section of  
the "Data Assessment Narrative".

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	YES	NO
A.1.10.2 Is correlation coefficient less than 0.995 for:		
Mercury Analysis?	<input type="checkbox"/>	<input type="checkbox"/>
Cyanide Analysis?		<input type="checkbox"/>
Atomic Absorption Analysis?	<input type="checkbox"/>	<input type="checkbox"/>

**ACTION:** If yes, flag the associated data as estimated.

**NOTE:** The data validator shall calculate the correlation  
coefficient using concentrations of the standards  
and the corresponding instrument response  
( e.g. absorbance, peak area, peak height, etc.).

A.1.10.3 In the instance where less than 4 standards are  
measured in absorbance (or peak area, peak height, etc.)  
mode, are the remaining standards analyzed in

concentration mode immediately after calibration  
within  $\pm 10\%$  of the true values? [ ]

**ACTION:** If no, flag the associated data as estimated  
if standards are not within  $\pm 10\%$  of true values.  
Do not flag the data as estimated in linear range  
indicated by good recovery of standard(s).

A.1.11 **Form II A (Initial and Continuing Calibration Verification)-**

A.1.11.1 Present and complete for every metal and cyanide? [ ] \_ \_

Present and complete for AA and ICP when both are  
used for the same analyte? [ ] \_ \_

**ACTION:** If no for any of the above, prepare Telephone  
Record Log and contact laboratory.

A.1.11.2 Circle on each Form IIA all percent recoveries that  
are outside the contract windows.  
Are all calibration standards (initial and continuing)  
within control limits:

Metals- 90-110%R? [ ] \_

Hg - 80-120%R? [ ] \_

Cyanides- 85-115%R? [ ]

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**ACTION:** Flag as estimated (J) all positive data (not  
flagged with a "U") analyzed between a  
calibration standard with %R between 75-89%  
(65-79% for Hg; 70-84% for CN) or 111-125%  
(121-135% for Hg; 116-130% for CN) recovery and  
nearest good calibration standard. Qualify results  
<IDL as estimated (UJ) if the ICV or CCV %R is  
75-89% (CN, 70-84% ; HG, 65-79%). Reject (red-line)  
as unacceptable data if recovery of the ICV or  
CCV is outside the range 75-125% (CN, 70-130%; Hg,  
65-135%). Qualify five samples on either side of

YES      NO

verification standard out of control limits.

A.1.11.3 Was continuing calibration performed every 10 samples  
or every 2 hours? ☐ ☐

Was ICV for cyanides distilled? ☐ ☐

**ACTION:** If no for any of the above, write in the  
Contract-Problem/Non-Compliance section of the  
"Data Assessment Narrative".

A.1.12 **Form II B (CRDL Standards for AA and ICP) -**

A.1.12.1 Was a CRDL standard (CRA) analyzed after initial  
calibration for all AA metals (except Hg)? ☐ ☐

Was a mid-range calib. verification standard distilled  
and analyzed for cyanide analysis? ☐ ☐

Was a 2xCRDL ( or 2xIDL when IDL>CRDL) analyzed (CRI)  
for each ICP run? ☐ ☐  
(Note: CRI for AL,Ba,Ca,Fe,Mg,Na,or K is not required.)

**ACTION:** If no for any of the above, flag as estimated  
all data falling within the affected ranges.  
The affected ranges are:  
AA Analysis - \*\*True Value  $\pm$  CRDL  
ICP Analysis - \*\*True Value  $\pm$  2CRDL  
CN Analysis - \*\*True Value  $\pm$  0.5 x True Value.

\*\*True value of CRA, CRI or mid-range standard. Substitute IDL for CRDL when IDL > C  
Compute the concentration of the missing mid-range standard from the calibration rang

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A.1.12.2 Was CRI analyzed after ICV/ICB and before the final

YES NO

CCV/CCB, and twice every eight hours of ICP run? [ ]

**ACTION:** If no, write in Contract Problem/Non-Compliance Section of the "Data Assessment Narrative".

A.1.12.3 Circle on each Form IIB all the percent recoveries that are outside the acceptance windows.

Are CRA and CRI standards within control limits:

Metals 80 - 120%R? [ ]

Is mid-range standard within control limits:

Cyanide 80 - 120%R? [ ]

**ACTION:** Flag as estimated all sample results within the affected range if the recovery of the standard is between 50-79%; flag only positive data within the affected range if the recovery is between 121-150%; reject all data within the affected range if the recovery is less than 50%; reject only positive data within the affected range if the recovery is greater than 150%. Qualify 50% of the samples on either side of CRI standard outside the control limits.

**Note:** Flag or reject the final results only when sample raw data are within the affected ranges and the CRDL standards are outside the acceptance windows.

A.1.13 **Form III (Initial and Continuing Calibration Blanks)**

A.1.13.1 Present and complete? [ ]

For both AA and ICP when both are used for the same analyte? [ ]

Was an initial calibration blank analyzed? [ ]

Was a continuing calibration blank analyzed after every 10 samples or every 2 hours (which ever is more frequent)? [ ]

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	<u>YES</u>	<u>NO</u>
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**ACTION:** If no, prepare Telephone Record Log, contact laboratory and write in the Contract-Problems/Non-Compliance section of the "Data Assessment Narrative".

A.1.13.2 Circle on each Form III all calibration blank values that are above CRDL (or 2 x IDL when IDL > CRDL).

Are all calibration blanks (when IDL<CRDL) less than or equal to the Contract Required Detection Limits (CRDLs)? [ ☐ ] ☐

Are all calibration blanks less than two times Instrument Detection Limit (when IDL>CRDL)? [ ☐ ] ☐

**ACTION:** If no for any of the above, flag as estimated (J) positive sample results when raw sample value is less than or equal to calibration blank value analyzed between calibration blank with value over CRDL (or 2xIDL) and nearest good calibration blank.  
Flag five samples on either side of the calibration blank outside the control limits.

A.1.14 **FORM III (Preparation Blank) -**  
(Note: The preparation blank for mercury is the same as the calibration blank.)

A.1.14.1 Was one prep. blank analyzed for:

each Sample Delivery Group (SDG)?	[ <input type="checkbox"/> ]	<input type="checkbox"/>
each batch of digested samples?	[ <input type="checkbox"/> ]	<input type="checkbox"/>
each matrix type?	[ <input type="checkbox"/> ]	<input type="checkbox"/>
both AA and ICP when both are used for the same analyte?	[ <input type="checkbox"/> ]	<input type="checkbox"/>

**ACTION:** If no for any of the above, flag as estimated (J) all the associated positive data <10 x IDLs for which prep. blank



was not analyzed.  
**NOTE:** If only one blank was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).

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A.1.14.2	Is concentration of prep. blank value greater than the CRDL when IDL is less than or equal to CRDL?		[ ]
	If yes, is the concentration of the sample with the least concentrated analyte less than 10 times the prep.blank?		[ ]
	<b><u>ACTION:</u></b> If yes, reject (red-line) all associated data greater than CRDL concentration but less than ten times the prep. blank value.		
A.1.14.3	Is concentration of prep. blank value (Form III) less than two times IDL, when IDL is greater than CRDL?	[ ]	
	<b><u>ACTION:</u></b> If no, reject (red-line) all positive sample results when sample raw data are less than 10 times the prep. blank value.		
A.1.14.4	Is concentration of prep. blank below the negative CRDL?		[ ]
	<b><u>ACTION:</u></b> If yes, reject (red-line) all associated sample results less than 10xCRDL.		
A.1.15	<b><u>Form IV (ICP Interference Check Sample)</u></b>		
A.1.15.1	Present and complete?	[ ]	
	(NOTE: Not required for furnace AA, flame AA, mercury, cyanide and Ca, Mg, K and Na.)		
	Was ICS analyzed at beginning and end of run (or at least twice every 8 hours)?	[ ]	

A.1.15.2 Circle all values on each Form IV that are more than  $\pm 20\%$  of true or established mean value.

If no, is concentration of Al, Ca, Fe, or Mg lower than the respective concentration in ICS? [ ]

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A.1.16 Form V A (Spiked Sample Recovery - Pre-Digestion/Pre-Distillation)-  
( **Note:** Not required for Ca, Mg, K, and Na (both matrices), Al, and Fe  
(soil only.)

**ACTION:** If no for any of the above, flag as estimated (J) all the positive data less than four times the spiking levels specified in SOW for which spiked sample was not analyzed.

**NOTE:** If one spiked sample was analyzed for more than 20 samples, then first 20 samples analyzed do not have to be flagged as estimated (J).

A.1.16.2 Was field blank used for spiked sample? \_\_\_\_\_ [ ] \_\_\_\_\_

**ACTION:** If yes, flag all positive data less than 4 x spike added as estimated (J) for which field blank was used as spiked sample.

A.1.16.3 Circle on each Form VA all spike recoveries that are outside control limits (75% to 125%).

Are all recoveries within control limits? [ ] \_\_\_\_\_

If no, is sample concentration greater than or equal to four times spike concentration? [ ] \_\_\_\_\_

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YES NO

**ACTION:** If yes, disregard spike recoveries for analytes whose concentrations are greater than or equal to four times spike added. If no, circle those analytes on Form V for which sample concentration is less than four times the spike concentration.

Are results outside the control limits (75-125%) flagged with "N" on Form I's and Form VA? [ ] \_\_\_\_\_

**ACTION:** If no, write in the Contract - Problem/Non - Compliance section of "Data Assessment Narrative".

A.1.16.4 Aqueous

Are any spike recoveries:

(a) less than 30%? \_\_\_\_\_ [ ] \_\_\_\_\_

(b) between 30-74%? [ ] \_\_\_\_\_

(c) between 126-150%? \_\_\_\_\_ [ ] \_\_\_\_\_

(d) greater than 150%? \_\_\_\_\_ ☐

**ACTION:** If less than 30%, reject all associated aqueous data; if between 30-74%, flag all associated aqueous data as estimated (J); if between 126-150%, flag as estimated (J) all associated aqueous data not flagged with a "U"; if greater than 150%, reject (red-line) all associated aqueous data not flagged with a "U".

A.1.16.5 **Soil/Sediment**

Are any spike recoveries:

(a) less than 10%? \_\_\_\_\_ ☐

(b) between 10-74%? \_\_\_\_\_ ☐

(c) between 126-200%? \_\_\_\_\_ ☐

(d) greater than 200%? \_\_\_\_\_ ☐

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	<u>YES</u>	<u>NO</u>
<b>ACTION:</b> If less than 10%, reject all associated data; if between 10-74%, flag all associated data as estimated; if between 126-200%, flag as estimated all associated data was not flagged with a "U"; if greater than 200%, reject all associated data not flagged with a "U".		

A.1.17 **Form VI (Lab Duplicates)**

A.1.17.1 Present and complete for: each SDG? ☐

each matrix type? ☐

each concentration range (i.e. low, med., high)? [ ]

both AA and ICP when both are used for the same  
analyte? [ ]

**ACTION:** If no for any the above, flag as estimated  
(J) all the data  $\geq$ CRDL\* for which duplicate  
sample was not analyzed.

**Note:** 1. If one duplicate sample was analyzed for  
more than 20 samples, then first 20 samples do not  
have to be flagged as estimated.  
2. If percent solids for soil sample and its duplicate  
differ by more than 1%, prepare a Form VI for each  
duplicate pair, report concentrations in ug/L  
on wet weight basis and calculate RPD or Difference  
for each analyte.

A.1.17.2 Was field blank used for duplicate analysis? [ ]

**ACTION:** If yes, flag all data  $\geq$ CRDL\* as estimated  
(J) for which field blank was used as duplicate.

A.1.17.3 Are all values within control limits (RPD 20% or  
difference  $\leq \pm$ CRDL)? [ ]

If no, are all results outside the control limits  
flagged with an \* on Form I's and VI? [ ]

**ACTION:** If no, write in the Contract - Problems/Non-  
Compliance section of "Data Assessment Narrative".

\* Substitute IDL for CRDL when IDL > CRDL.

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YES NO

**NOTE:** 1. RPD is not calculable for an analyte of the  
sample - duplicate pair when both values are  
less than IDL.  
2. If the result of lab duplicate analyzed  
by GFAA is rejectable due to coefficient of

correlation of MSA, analytical spike recovery,  
or duplicate injections criteria, do not apply  
precision criteria to metals analyzed by GFAA.

A.1.17.4 Aqueous

Circle on each Form VI all values that are:

RPD > 50%, or  
Difference > CRDL\*

Is any RPD greater than 50% where sample and duplicate  
are both greater than or equal to 5 times \*CRDL? \_\_\_\_\_ [\_\_\_\_]

Is any difference\*\* between sample and duplicate greater  
than \*CRDL where sample and/or duplicate is less than  
5 times \*CRDL? \_\_\_\_\_ [\_\_\_\_]

**ACTION:** If yes, flag the associated data as estimated.

A.1.17.5 Soil/Sediment

Circle on each Form VI all values that are:

RPD > 100%, or  
Difference > 2 x CRDL\*

Is any RPD (where sample and duplicate are both  
greater than or equal to 5 times \*CRDL) :  
\_\_\_\_\_ > 100%? \_\_\_\_\_ [\_\_\_\_]

Is any \*\*difference between sample and duplicate  
(where sample and/or duplicate is less than 5x\*CRDL) :  
\_\_\_\_\_ > 2x\*CRDL? \_\_\_\_\_ [\_\_\_\_]

\* Substitute IDL for CRDL when IDL > CRDL.

\*\* Use absolute values of sample and duplicate to calculate the difference.

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YES

NO

**ACTION:** If yes, flag the associated data as estimated.

A.1.18 **Field Duplicates**

A.1.18.1 Were field duplicates analyzed? [ ]

**ACTION:** If yes, prepare a Form VI for each aqueous field duplicate pair. Prepare a Form VI for each soil duplicate pair, if percent solids for sample and its duplicate differ by more than 1%; report concentrations of soils in ug/l on wet weight basis and calculate RPDs or Difference for each analyte.

**NOTE:** 1. Do not calculate RPD when both values are less than IDL.  
2. Flag all associated data only for field duplicate pair.

A.1.18.2 **Aqueous**

Circle all values on self prepared Form VI for field duplicates that are:

RPD > 50%, or  
Difference > CRDL\*

Is any RPD greater than 50% where sample and duplicate are both greater than or equal to 5 times \*CRDL? [ ]

Is any \*\*difference between sample and duplicate greater than \*CRDL where sample and/or duplicate is less than 5 times \*CRDL? [ ]

**ACTION:** If yes, flag the associated data as estimated.

\* Substitute IDL for CRDL when IDL > CRDL.

\*\* Use absolute values of sample and duplicate to calculate the difference.

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YES NO

A.1.18.3 Soil/Sediment

Circle all values on self prepared Form VI for  
field duplicates that are:

RPD >100%, or

Difference > 2 x CRDL\*

Is any RPD (where sample and duplicate are both  
greater than 5 times \*CRDL) :

>100%? ☐ ☐

Is any \*\*difference between sample and duplicate  
(where sample and/or duplicate is less than 5x \*CRDL) :

>2x \*CRDL? ☐ ☐

**ACTION:** If yes, flag the associated data as estimated.

A.1.19 Form VII (Laboratory Control Sample) (Note: LCS - not  
required for aqueous Hg and cyanide analyses.)

A.1.19.1 Was one LCS prepared and analyzed for:

each SDG? ☐ ☐

each batch samples digested/distilled? ☐ ☐

both AA and ICP when both are used for the same  
analyte? ☐ ☐

**ACTION:** If no for any of the above, prepare Telephone  
Record Log and contact laboratory for submittal



of results of LCS. Flag as estimated (J) all the data for which LCS was not analyzed.

**NOTE:** If only one LCS was analyzed for more than 20 samples, then first 20 samples close to LCS do not have to be flagged as estimated.

\* Substitute IDL for CRDL when IDL > CRDL.

\*\* Use absolute values of sample and duplicate to calculate the difference.

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YES      NO

#### A.1.19.2      Aqueous LCS

Circle on each Form VII the LCS percent recoveries outside control limits (80 - 120%) except for aqueous Ag and Sb.

Is any LCS recovery:	less than 50%?	_____	[ <input type="checkbox"/> ]
	between 50% and 79%?		[ <input type="checkbox"/> ]
	between 121% and 150%?	_____	[ <input type="checkbox"/> ]
	greater than 150%?		[ <input type="checkbox"/> ]

**ACTION:** Less than 50%, reject (red-line) all data; between 50% and 79%, flag all associated data as estimated (J); between 121% and 150%, flag all positive (not flagged with a "U") results as estimated; greater than 150%, reject all positive results.

#### A.1.19.3      Solid LCS

**NOTE:** 1. If "Found" value of LCS is rejectable due to duplicate injections or analytical spike recovery criteria, regardless of LCS recovery, flag the associated data

- as estimated (J).
2. If IDL of an analyte is equal to or greater than true value of LCS, disregard the "Action" below even though LCS is out of control limits.

Is LCS "Found" value higher than the control limits on Form VII? [    ]

**ACTION:** If yes, qualify all associated positive data as estimated.

Is LCS "Found" value lower than the Control limits on Form VII? \_\_\_\_\_ [    ]

**ACTION:** If yes, qualify all associated data as estimated.

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YES      NO

A.1.20 **Form IX (ICP Serial Dilution) -**

**NOTE:** Serial dilution analysis is required only for initial concentrations equal to or greater than 10 x IDL.

A.1.20.1 Was Serial Dilution analysis performed for:

each SDG? [    ] \_\_\_\_\_

each matrix type? [    ] \_\_\_\_\_

each concentration range (i.e. low, med.)? [    ]

**ACTION:** If no for any of the above, flag as estimated all the positive data  $\geq 10 \times \text{IDLs}$  or  $\geq \text{CRDL}$  when  $10 \times \text{IDL} \leq \text{CRDL}$  for which Serial Dilution Analysis was not performed.

A.1.20.2 Was field blank(s) used for Serial Dilution Analysis? ☐ ☐

**ACTION:** If yes, flag all associated data  $\geq 10 \times \text{IDL}$  as estimated (J). If  $10 \times \text{IDL} \leq \text{CRDL}$ , flag all data  $\geq \text{CRDL}$ .

A.1.20.3 Are results outside control limit flagged with an "E" on Form I's and Form IX when initial concentration on Form IX is equal to 50 times IDL or greater. ☐

**ACTION:** If no, write in the Contract-Problem/Non-Compliance section of the "Data Assessment Narrative".

A.1.20.4 Circle on each Form IX all percent difference that are outside the control limits for initial concentrations equal to or greater than  $10 \times \text{IDL}$ s only.

Are any % difference values:

> 10%? ☐

$\geq 100\%$ ? ☐

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**ACTION:** Flag as estimated (J) all the associated sample data  $\geq 10 \times \text{IDL}$ s (or  $\geq \text{CRDL}$  when  $10 \times \text{IDL} \leq \text{CRDL}$ ) for which percent difference is greater than 10% but less than 100%. Reject (red-line) all the associated sample results equal to or greater than  $10 \times \text{IDL}$ s (or  $\geq \text{CRDL}$  when  $10 \times \text{IDL} \leq \text{CRDL}$ ) for which PD is greater than or equal to 100%.

YES

NO

**Note:** Flag or reject on Form I's only the sample results whose associated raw data are  $\geq 10 \times \text{IDL}$  (or  $\geq \text{CRDL}$  when  $10 \times \text{IDL} \leq \text{CRDL}$ )

A.1.21 Furnace Atomic Absorbtion (AA) OC Analysis

A.1.21.1 Are duplicate injections present in furnace raw data (except during full Method of Standard Addition) for each sample analyzed by GFAA? ☐ ☐

**ACTION:** If no, reject the data on Form I's for which duplicate injections were not performed.

A.1.21.2 Do the duplicate injection readings agree within 20% Relative Standard Deviation (RSD) or Coefficient of Variation (CV) for concentration greater than CRDL? ☐ ☐

Was a dilution analyzed for sample with analytical spike recovery less than 40%? ☐ ☐

**ACTION:** If no for any of the above, flag all the associated data as estimated.

A.1.21.3 Is \*analytical spike recovery outside the control limits (85-115%) for any sample? ☐ ☐

**ACTION:** If yes, flag as estimated the affected sample results if the recovery is between 10-84%; if the recovery is between 115-200%, flag the associated positive sample results as estimated; reject the associated sample results if the recovery is less than 10%; reject positive sample results if the recovery is greater than 200%.

\* Analytical spike is not required on the pre-digestion spiked sample.

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YES

NO

**NOTE:** Reject or flag the data only when the affected sample(s) was not subsequently analyzed by Method of Standard Addition.

A.1.22 Form VIII (Method of Standard Addition Results)

A.1.22.1 Present? [ ]

If no, is any Form I result coded with "S" or a "+"? [ ]

**ACTION:** If yes, write request on Telephone Record Log  
and contact laboratory for submittal of Form VIII.

A.1.22.2 Is coefficient of correlation for MSA less than 0.990 for  
any sample? [ ]

**ACTION:** If yes, reject (red-line) the affected data.

A.1.22.3 Was \*MSA required for any sample but not performed? [ ]

Is coefficient of correlation for MSA less than 0.995? [ ]

Are MSA calculations outside the linear range of the  
calibration curve generated at the beginning of the  
analytical run? [ ]

**ACTION:** If yes for any of the above, flag all  
the associated data as estimated (J).

A.1.22.4 Was proper quantitation procedure followed correctly  
as outlined in the SOW on page E-23? [ ]

**ACTION:** If no, note exception under Contract Problem/  
Non-Compliance section of the "Data Assessment  
Narrative", and prepare a separate list.

\* MSA is not required on LCS and prep. blank.

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YES

NO

A.1.23 **Dissolved/Total or Inorganic/Total Analytes -**

A.1.23.1 Were any analyses performed for dissolved as well as total analytes on the same sample(s). [    ]

Were any analyses performed for inorganic as well as total (organic + inorganic) analytes on the same sample(s)? [    ]

**NOTE:** 1. If yes, prepare a list comparing differences between all dissolved (or inorganic) and total analytes. Compute the differences as a percent of the total analyte only when dissolved concentration is greater than CRDL as well as total concentration.  
2. Apply the following questions only if inorganic (or dissolved ) results are (i) above CRDL, and (ii) greater than total constituents.  
3. At least one preparation blank, ICS, and LCS should be analyzed in each analytical run.

A.1.23.2 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 10%? \_\_\_\_\_ [    ] \_\_\_\_\_

A.1.23.3 Is the concentration of any dissolved (or inorganic) analyte greater than its total concentration by more than 50%? \_\_\_\_\_ [    ] \_\_\_\_\_

**ACTION:** If more than 10%, flag both dissolved (or inorganic) and total values as estimated (J); if more than 50%, reject (red-line) the data for both values.

A.1.24 **Form I (Field Blank) -**

**(Note: Designate "Field Blank" as such on Form I.)**

A.1.24.1 Circle all field blank values on Form I that are greater than CRDL, (or 2 x IDL when IDL > CRDL).

Is field blank concentration less than CRDL (or 2 x IDL when IDL > CRDL) for all parameters

of associated aqueous and soil samples?  
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\_\_\_\_\_  
If no, was field blank value already rejected  
due to other QC criteria?

YES NO

[ ]

**ACTION:** If no, reject (except field blank results)  
all associated positive sample data less  
than or equal to five times the field blank  
value. Reject on Form I's the soil sample  
results that when converted to ug/L on wet  
basis are less than or equal to five times  
the field blank value in ug/L.

A.1.25 **Form X, XI, XII (Verification of Instrumental Parameters).**

A.1.25.1 Is verification report present for:

Instrument Detection Limits (quarterly)? [ ] \_\_\_\_

ICP Interelement Correction Factors (annually)? [ ]

ICP Linear Ranges (quarterly)? [ ] \_\_\_\_

**ACTION:** If no, contact TPO of the lab.

A.1.25.2 **Form X (Instrument Detection Limits)** - (Note: IDL is not  
required for Cyanide.)

A.1.25.2.1 Are IDLs present for: all the analytes? [ ] \_\_\_\_

all the instruments used? [ ] \_\_\_\_

For both AA and ICP when both are used for the same  
analyte? [ ]

**ACTION:** If no for any of the above, prepare  
Telephone Record Log and contact

laboratory.

A.1.25.2.2 Is IDL greater than CRDL for any analyte?

[   ]

If yes, is the concentration on Form I of the sample analyzed on the instrument whose IDL exceeds CRDL, greater than 5 x IDL.

[   ]

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YES      NO

**Action :** If no, flag as estimated all values less than five times IDL of the instrument whose IDL exceeds CRDL.

A.1.25.3 Form XI (Linear Ranges)

A.1.25.3.1 Was any sample result higher than high linear range of ICP.

\_\_\_\_ [   ] \_\_\_\_

Was any sample result higher than the highest calibration standard for non-ICP parameters?

\_\_\_\_ [   ] \_\_\_\_

If yes for any of the above, was the sample diluted to obtain the result on Form I?

[   ]

**ACTION:** If no, flag the result reported on Form I as estimated(J).

A.1.26 Percent Solids of Sediments

A.1.26.1 Are percent solids in sediment(s):

< 50%?

\_\_\_\_ [   ] \_\_\_\_

< 10%?

\_\_\_\_ [   ] \_\_\_\_



**ACTION:** If yes, qualify as estimated all the results of a sample that has per cent solids between 10%-50% (i.e. moisture content between 50%-90%). Reject all the results of a sample that has per cent solids less than 10% (i.e. moisture content greater than 90%).

**NOTE:** Reject or flag(J) only the sample results that were not previously rejected or flagged due to other QC criteria.

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Case#	_____	Site	_____	Matrix: Soil	_____
SDG#	_____	Lab	_____	Water	_____
Contractor	_____	Reviewer	_____	Other	_____

A.2.1 **Validation Flags-** The following flags have been applied in red by the data validator and must be considered by the data user.

J- This flag indicates the result qualified as **estimated**

Red- Line- A red-line drawn through a sample result indicates **unusable** value. The red-lined data are known to contain significant errors based on documented information and must not be used by the data user.

**Fully Usable Data-** The results that do not carry "J" or "red-line" are fully usable.

Contractual Qualifiers- The legend of contractual qualifiers applied by the 1a on Form I's is found on page B-20 of SOW ILM01.0.

A.2.2 The data assessment is given below and on the attached sheets.

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A.2.2 (continuation)

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A.2.3 Contract-Problem/Non-Compliance

MMB/ESAT Rviewer: \_\_\_\_\_ Date: \_\_\_\_\_  
Signature

Contractor Reviewer: \_\_\_\_\_ Date: \_\_\_\_\_  
Signature

Verified by: \_\_\_\_\_ Date: \_\_\_\_\_

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(SMO Report)

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CONTRACT NON-COMPLIANCE  
(SMO REPORT)

Regional Review of Uncontrolled Hazardous Waste  
Site Contract Laboratory Data Package

CASE NO. \_\_\_\_\_

The hardcopied (laboratory name)

Inorganic data package received at Region II has been reviewed and the quality assurance performance data summarized. The data reviewed included:

SMO Sample No.: \_\_\_\_\_

Conc. & Matrix: \_\_\_\_\_

Contract No. (\_\_\_\_\_) requires that specific analytical work be done and that associated reports be provided by the contractor to the Regions, EMSL-LV, and SM general criteria used to determine the performance were based on an examination of:

- |                                 |                              |
|---------------------------------|------------------------------|
| - Data Completeness             | - Duplicate Analysis Results |
| - Matrix Spike Results          | - Blank Analysis Results     |
| - Calibration Standards Results | - MSA Results                |

Items of non-compliance with the above contract are described below.

Comments: \_\_\_\_\_

\_\_\_\_\_

\_\_\_\_\_

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\_\_\_\_\_  
Reviewer's Initial

\_\_\_\_\_  
Date

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Summary Form (Inorganics)

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## Inorganic Analysis

Region

SITE

NO. OF SAMPLES/  
MATRIX

REVIEWER (IF NOT ESD)

REVIEWER'S NAME

DPO: ACTION \_\_\_\_\_ FYI \_\_\_\_\_ COMPLETION DATE \_\_\_\_\_

DATA ASSESSMENT SUMMARY

		ICP	AA	Hg	CYANIDE
1.	HOLDING TIMES	_____	_____	_____	_____
2.	CALIBRATIONS	_____	_____	_____	_____
3.	BLANKS	_____	_____	_____	_____
4.	ICS	_____	_____	_____	_____
5.	LCS	_____	_____	_____	_____
6.	DUPLICATE ANALYSIS	_____	_____	_____	_____
7.	MATRIX SPIKE	_____	_____	_____	_____
8.	MSA	_____	_____	_____	_____
9.	SERIAL DILUTION	_____	_____	_____	_____
10.	SAMPLE VERIFICATION	_____	_____	_____	_____
11.	OTHER QC	_____	_____	_____	_____
12.	OVERALL ASSESSMENT	_____	_____	_____	_____

O = Data has no problems/or qualified due to minor problems.

M = Data qualified due to major problems.

Z = Data unacceptable.

X = Problems, but do not affect data.

ACTION ITEMS: \_\_\_\_\_

AREAS OF CONCERN: \_\_\_\_\_

NOTABLE PERFORMANCE: \_\_\_\_\_